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METHODS FOR MEASUREMENT OF AIR POLLUTION

PART XVII C_1 TO C_5 HYDROCARBONS IN AIR BY GAS CHROMATOGRAPHY

(First Reprint MARCH 1993)

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NEW DELHI 110002

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Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART XVII C_1 TO C_5 HYDROCARBONS IN AIR BY GAS CHROMATOGRAPHY

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Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART XVII C_1 TO C_5 HYDROCARBONS IN AIR BY GAS CHROMATOGRAPHY

0. FOREWORD

0.1 This Indian Standard (Part XVII) was adopted by the Indian Standards Institution on 25 March 1979, after the draft finalized by the Air Pollution Sectional Committee had been approved by the Chemical Division Council.

0.2 The measurement of C_1 to C_5 hydrocarbons provides data for assessing atmospheric hydrocarbon pollution from motor vehicles, filling stations, petroleum refineries, and a variety of industrial processes employing solvents. The method given in this standard is suitable for determining photochemically reactive hydrocarbons in the atmosphere, the hydrocarbon contamination in breathing air tanks, and in work-room atmospheres. Because ethylene is phytotoxic and the olefins are generally photochemically reactive, a gas chromatographic column is desirable that resolves completely all components of an air sample up to a reasonably high carbon number. The effort to obtain atmospheric hydrocarbon data is based upon the desire to accumulate information concerning prevalent regional air quality, thus requiring routine sampling and analysis. This method meets the requirements of resolution, sensitivity, and operational efficiencies.

0.3 In the preparation of this standard, considerable assistance has been derived from ASTM D2820-72 ' Standard test method for C_1 through C_5 hydrocarbons in the atmosphere by gas chromatography ' published by American Society for Testing and Materials, USA.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the method of measurement of the concentration of individual C_1 to C_5 hydrocarbons in the atmosphere. The lower limit of measurement is 0·01 parts per million (ppm) by volume (that is, 0·01μl/l). For analysis without concentration of the sample, the elution of the 17 hydrocarbons as given in Table 1 is accomplished within approximately 16 minutes. Hydrocarbons of higher molecular mass than C_5 may be measured by concentration of the air sample.

1.2 The lower limit of measurement of the C_3 to C_5 hydrocarbons may be extended to 0·1 parts per billion (ppb) by concentrating 100 ml of gas sample in a freeze trap. Methane is not collected quantitatively in the freeze-trap and the resolution of ethane and ethylene chromatographic peaks is reduced in the concentrated sample.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 4167-1966* shall apply.

3. OUTLINE OF THE METHOD

3.1 This is a rapid method intended for routine sampling and analysis. Directions are given for the collection of grab samples and integrated samples. An air sample is collected in a sealed plastic bag fitted with an air valve. The sample is delivered to the laboratory where the C_1 to C_5 hydrocarbons can be identified and quantitated to 0·01 ppm by analysis of 1 ml of air sample without concentration. The C_3 to C_5 hydrocarbons may be quantitated to 0·1 ppb by concentrating 100 ml of air sample in a freeze trap immersed in liquid oxygen, dry ice or a mixture of dry ice and acetone. Liquid nitrogen is not used, to avoid condensation of oxygen, in freezing out the air sample.

NOTE — Liquid oxygen should not be allowed to come into contact with oil, grease or carbonaceous materials of any kind, because liquid oxygen can promote intense combustion. Immediately remove any clothing that becomes saturated with liquid oxygen.

3.2 The gas chromatographic analysis is performed on a single packed column, operated isothermally at 0° C. Flame ionization is used for detection with pure oxygen supplied to the hydrogen flame to enhance its sensitivity.

3.3 2,2-dimethylpropane and trans-2-butene are the only known compounds that elute from the columns together under conditions of the method.

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*Glossary of terms relating to air pollution.

4. INTERFERENCES

4.1 Generally, for a homogeneous, urban air mass, hydrocarbon contaminants would be expected to fall within the ranges given in Table 1. Average losses of C_1 to C_5 atmospheric hydrocarbons in these bags are known to be less than 6 percent for up to 3 days' storage. The bags should preferably be stored below 27°C. If there are large deviations from the extremes of the range, a critical review of the data should be made. The review should consider contamination of the sample bag or sample valve and lines by atmospheres containing high hydrocarbon concentrations.

4.2 As a general rule, if methane, ethane or propane concentrations are high relative to other identified hydrocarbons, look for local source emissions of natural gas. If acetylene and the butenes are high, look for a nearby automobile exhaust source. If *n*-butane, *n*-pentane, and *iso*-pentane are high, look for a source of gasoline evaporation.

TABLE 1 RANGES OF HYDROCARBON VALUES EXPECTED IN URBAN AIR MASS

(*Clauses* 1.1 and 4.1)

COMPONENT	RANGE, ppm IN AIR	
	Min	*Max*
(1)	(2)	(3)
Methane	1·2	15
Ethane	0·005	0·5
Ethylene	0·004	0·3
Propane	0·003	0·3
Propene	0·001	0·1
Acetylene	0·000	0·2
*Iso*butane	0·001	0·1
n-Butane	0·004	0·4
1-Butene	0·000	0·02
2-Methylpropene	0·000	0·02
2,2-Dimethylpropane	0·000	0·01
trans-2-butene	0·000	0·01
cis-2-butene	0·000	0·01
Iso-pentane	0·002	0·2
1-1,3-Butadiene	0·000	0·01
n-Pentane	0·002	0·2
3-Methyl-1-butene	0·000	0·01

5. APPARATUS

5.1 Gas Chromatography Assembly — A gas chromatograph assembly is shown in Fig. 1. Operating conditions are specified for a typical instrument. Equivalent apparatus may be substituted with the adjustment of operating conditions to provide the required performance. The principal components are given in **5.1.1** to **5.6.10**.

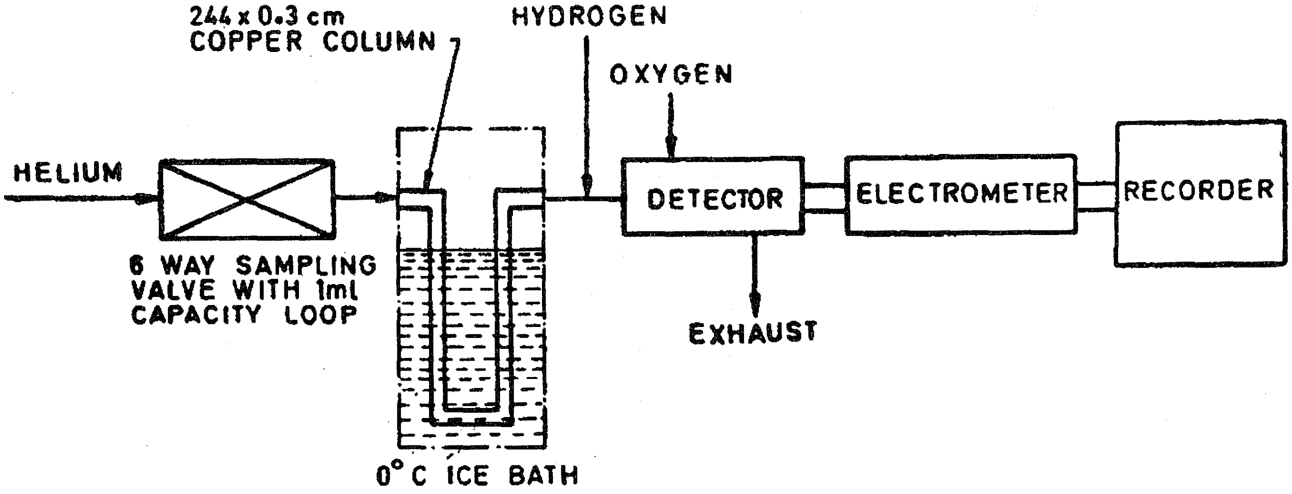

FIG. 1 GAS CHROMATOGRAPH ASSEMBLY FOR MEASURING C_1 TO C_5 ATMOSPHERIC HYDROCARBONS

5.1.1 The chromatography column is prepared according to directions given in **6.8**.

5.1.2 The specification of the sample injector depends upon whether analysis is to be made of 1 ml of the air sample directly or 100 ml of air sample is to be concentrated in a freeze-trap before analysis.

5.1.3 For direct analysis, the sample injector consists of a six-way sampling valve with a 1-ml stainless steel sampling loop.

5.1.4 When 100 ml of the air sample are concentrated, a freeze-trap is substituted for the sampling loop. Directions for preparation of the freeze trap are given in **6.9**.

5.1.5 *Dewar Flask* — 250-ml, for use with the freeze-trap.

5.1.6 *Gastight Syringe* — 100-ml.

5.1.7 *Water Bath* — Room-temperature, for defrosting the freeze-trap.

5.1.8 *Hydrogen Flame Ionization Detector*

5.1.9 *Electrometer* (*see* **5.6.8**).

5.1.10 *Strip Chart Recorder* — with a range from 0 to 1 mV and 1-s response, to measure detector response.

5.1.11 *Bath* — for thermostating the column at 0°C.

5.2 Air samples are collected and calibration standards are prepared in aluminized plastics bags. The bags may also be fabricated in the laboratory from the materials given in **5.2.1** to **5.2.4**.

5.2.1 *Polyester Film* — heat sealable, aluminized.

5.2.2 *Heat-Sealing Iron* — with a TFE-fluorocarbon cover.

5.2.3 *Clamp-on Valves, and Neoprene Gaskets*

5.2.4 *Punch* — for punching a 9 mm hole into the film into which the clamp-on valve is fitted.

5.3 The assembly of apparatus for preparing calibration standards is shown in Fig. 2. The principal components are given in **5.3.1** to **5.3.4**.

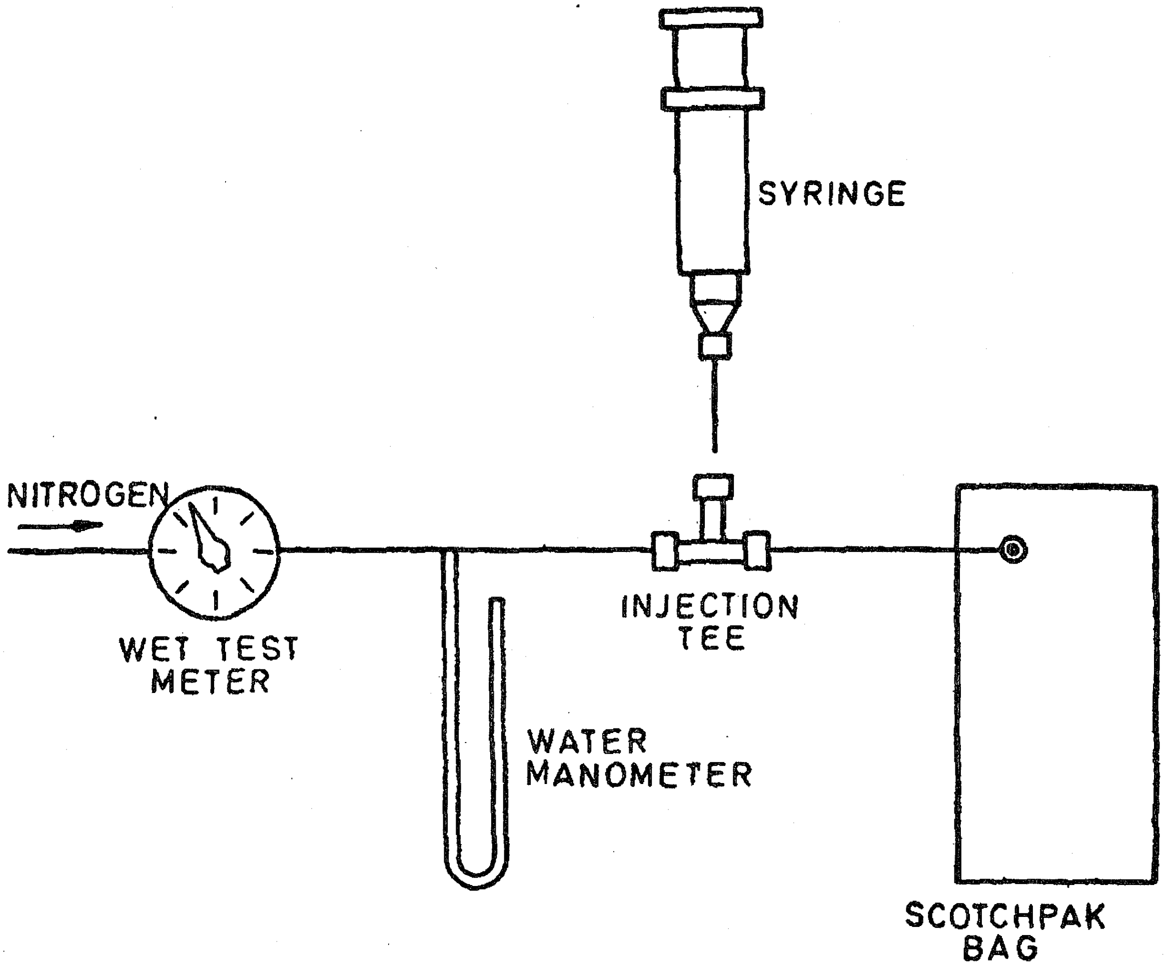

FIG. 2 ASSEMBLY FOR PREPARATION OF CALIBRATION STANDARDS

5.3.1 *Precision Wet Test Gas Meter*

5.3.2 *Stainless Steel Injection Tee* — with the centre arm fitted with a silicone rubber injection gasket.

5.3.3 *Gastight Syringes* — 10-ml and 50-ml.

5.3.4 *Aluminized Plastics Bags* — Two sizes : approximately 40-litre and 10-litre capacity. Recommended sizes are 460 × 1 220 mm and 460 × 310 mm respectively.

5.4 The following equipment is used for collecting grab samples of air.

5.4.1 *Aluminized Plastics Bags* — fitted with air valves, of approximately 2-litre capacity.

5.4.2 *Atomizer Rubber Bulb Set or Automatic Burette Bulb*

5.5 The assembly of equipment for collecting integrated samples is shown in Fig. 3. The principal components are given in **5.5.1** to **5.5.7**.

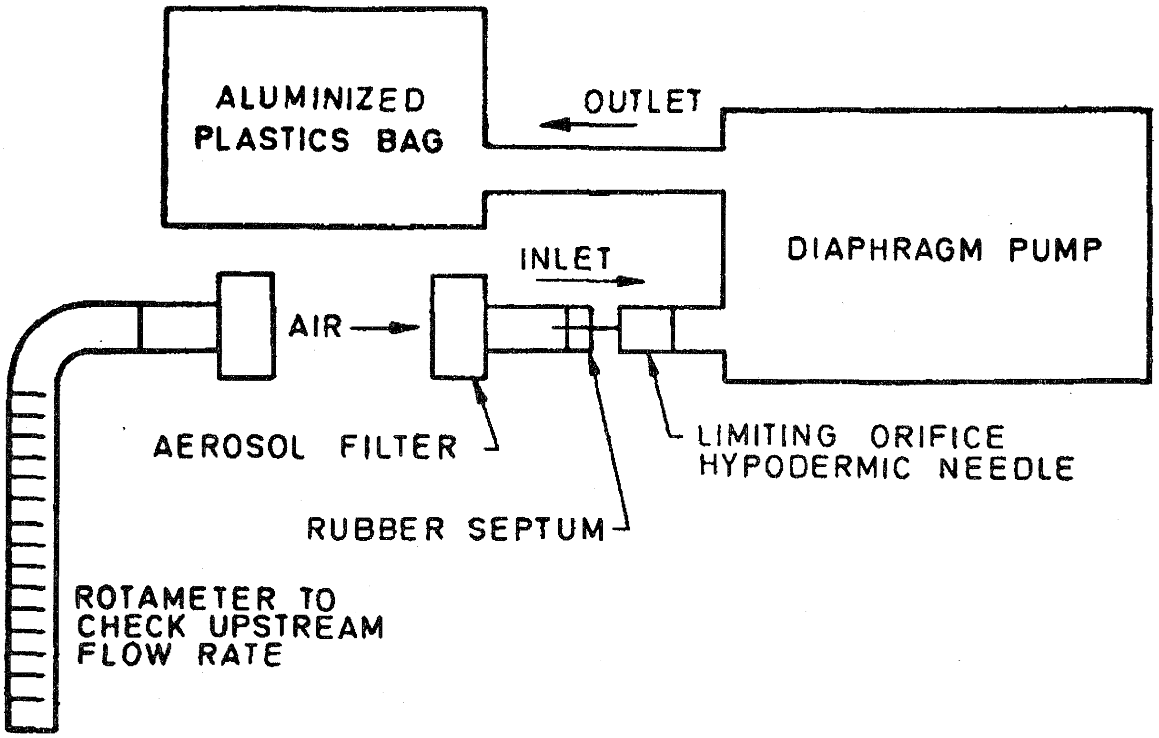

FIG. 3 ASSEMBLY FOR INTEGRATED HYDROCARBON SAMPLING

5.5.1 *Air-Sampling Pump* — diaphragm-actuated, capable of providing constant flow up to 10 l/min at 10^5 Pa and 0·066 l/min at 0·4 × 10^5 Pa.

5.5.2 *Aluminized Plastics Bags* — fitted with air valves : for 15-litre sample; for 30-litre samples. Recommended sizes are 460 × 460 mm and 460 × 920 mm respectively.

5.5.3 *Air-Metering Device* — A limiting orifice prepared from a hypodermic needle may also be used as an air-metering device.

5.5.4 *Electric Timer* — which automatically shuts off the sampling pump at the end of the sampling interval.

5.5.5 *Rubber Septum* — when limiting orifice needles are used. A sleeve-type serum bottle stopper is suitable.

5.5.6 *Flowmeter* — with the range from 1 to 85 ml/min at 100 k Pa and 21°C.

5.5.7 *Filter System* — upstream of the pump, is recommended for the entrapment of particulate matter (membrane filter or equivalent filter which does not absorb organic materials).

5.6 Instrument parameters defining the operating conditions for this method are as given in **5.6.1** to **5.6.10**.

5.6.1 *Column* — 244 cm long, 1·5 mm inside diameter, 17 mass percent oxydipropionitrile on activated alumina, 100 to 150 micron size.

5.6.2 *Temperature:*

Sample inlet system	room temperature
Detector	room temperature
Column	isothermal, 0°C

5.6.3 *Carrier Gas* — Helium or hydrogen at 30 ml/min and 560 k Pa.

5.6.4 *Detector Gas* — Hydrogen flow to the detector maintained at 28 ml/min by means of a restricted stainless steel capillary. Oxygen flow at 300 ml/min.

5.6.5 *Recorder Range* — 0 to 1 mV and 1-s response.

5.6.6 *Sample Size* — 1 ml for direct analysis; 100 ml for freeze trap.

5.6.7 *Freeze-Trap Flow* — Approximately 25 ml/min.

5.6.8 *Electrometer* — Sensitivity of 3×10^{-12}. A full scale on 1-mV recorder, min.

5.6.9 *Electrometer* — Attenuation of 10 × for methane, 1 × for the other hydrocarbons in atmospheric air samples.

5.6.10 *Detector Sensitivity* — 0·02°C/g with oxygen as combustion gas, min.

6. QUALITY OF REAGENTS

6.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977*) freshly boiled and cooled, shall be employed.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

6.2 The materials given in **6.2.1** to **6.2.4** shall be used for preparation of the chromatograph column.

6.2.1 *Alumina* (Al_2O_3) — 100 to 150 micron size, chromatographic grade.

6.2.2 *β, β-oxydiproprionitrile*

6.2.3 *Copper or Stainless Steel Tubing* — 3 mm outside diameter and 1·5 mm inside diameter.

6.2.4 *Dichloromethane*

6.3 The materials given in **6.3.1** to **6.3.3** shall be used for preparation of the freeze-trap.

6.3.1 *Chromosorb P* — 250 to 350 micron size.

6.3.2 *Stainless Steel Tubing* — 3 mm outside diameter and 2 mm inside diameter.

6.3.3 *Two On-off Gastight Toggle Valves* — to be fitted to the stainless steel tubing.

6.4 The gases given in **6.4.1** and **6.4.2** shall be supplied to the hydrogen flame detector.

6.4.1 *Hydrogen* — pure grade, 99·8 percent, free from water and organic material.

6.4.2 *Oxygen* — commercial grade 99·6 percent min.

6.5 *Helium* — reactor grade shall be used as the column, carrier gas, 99·995 percent *Min.* In case helium is not available hydrogen, pure grade, 99·8 percent, free from water and organic material may be used as column carrier gas.

6.6 The gases given in **6.6.1** and **6.6.2** shall be used for preparation of the calibration standards.

6.6.1 *Hydrocarbons* — pure grade, having a guaranteed minimum purity of 99 mole percent.

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*Specification for water for general laboratory use (*second revision*).

6.6.2 *Nitrogen* — pure grade, 99·9 percent min.

6.7 Liquid oxygen is required when the freeze trap is used. In case a liquid oxygen is not available, dry ice or a mixture of dry ice and acetone may be used.

6.8 The chromatograph column consists of 17 mass percent of β, β-oxydipropionitrile on activated alumina.

6.8.1 Place about 25 to 30 g of alumina in a 15 cm porcelain evaporating dish and wash several times with deionized water. After the excess water has been poured off, activate the alumina at 400°C for 9 h. Allow to cool in a desiccator for about 1 h. Weigh out 16·6 g of the activated alumina and pour immediately into another 15 cm porcelain evaporating dish containing 3·4 g of β, β-oxydipropionitrile dissolved in 40 ml of dichloromethane. Evaporate the solvent under a reflector infrared heat lamp with frequent stirring; remove residual solvent at 70°C at about 30 kPa for 5 h.

6.8.2 Fill a column by gravity flow and continual tapping with the column packing (*see* **6.8.1**). Coil and condition the column overnight at room temperature with carrier gas at 30 ml/min and inlet pressure 560 kPa.

6.9 The freeze-trap consists of a stainless steel tubing packed with Chromosorb P. Each end of the U-tube is fitted with a toggle valve.

7. SAMPLING

7.1 Grab samples shall be collected as described in **7.1.1** to **7.1.4**.

7.1.1 Flush the aluminized plastics bag three times with the ambient air to be sampled. This is done with a rubber burette bulb connected to the valve of the bag.

7.1.2 Fill the bag approximately three fourths full, close the air valve securely, and remove the burette bulb. Do not fill the bag to capacity. Some space should be allowed for expansion due to temperature and pressure variations.

7.1.3 Give the bag an identifying number and record appropriate field information, for example, date, time and location of sample collection; weather, traffic, air pollution conditions, etc.

7.1.4 The samples should be sent to the laboratory and analyzed as soon as possible (*see* **4.1** for the storability of C_1 to C_5 paraffinic and olefinic hydrocarbons in these aluminized plastics bags).

7.2 The equipment for collecting integrated samples is assembled as shown in Fig. 3.

7.2.1 The air flow into the aluminized plastics bag is measured by attaching a flowmeter to the upstream side of the diaphragm pump. If a micrometering valve is used instead of a limiting orifice, the valve is adjusted to the desired flow rate.

7.2.2 When the desired flow rate is obtained, disconnect the flowmeter. Set the electric timer for the period for which the sample is to be collected.

7.2.3 Near the end of the sampling period, measure the flow rate again.

7.2.4 Identify the bag sample and record appropriate field data as outlined in **7.1.3**.

7.2.5 It is not important to know the exact volume of the sample collected. A 1-ml portion of the sample is taken for gas chromatographic analysis and the concentration of hydrocarbons in the sample is based on 1 ml. It is important that a constant flow rate be maintained during the sampling period or the change in flow rate during the period be measured in order to ensure a valid integrated sample.

8. CALIBRATION

8.1 Calibration Standards — A calibration standard should be prepared for each of the hydrocarbons to be measured. Retention time is used for identification of the hydrocarbon and either peak height or peak area is used for quantitation of the hydrocarbon. Standard calibration mixtures using the assembly of equipment shown in Fig. 2 are prepared as given in **8.1.1** to **8.1.5**.

8.1.1 Purge the wet test meter with nitrogen for half an hour.

8.1.2 Connect the plastics bag to the system and start metering nitrogen into the bag. 40-litre bags are used for the 40-litre dilution; 10-litre bags are used for the 10-litre dilutions.

8.1.3 Inject the predetermined amount of each hydrocarbon into the nitrogen stream through the septum in the stainless steel tee. A 50-ml, gastight syringe for volumes greater than 10 ml; a 10-ml gastight syringe for volumes less than 10 ml.

8.1.4 To prepare standards containing less than 10 ppm of a hydrocarbon, a double dilution is required. First, a 1 000-ppm standard is prepared. Aliquots of this standard are diluted to produce standards with less than 10 ppm hydrocarbon (*see* Table 2).

TABLE 2 DILUTIONS FOR PREPARATION OF CALIBRATION STANDARDS

(*Clause* 8.1.4)

HYDROCARBON CONCENTRATION ppm (1)	ALIQUOT OF HYDROCARBON (2)	FINAL DILUTION VOLUME, litres (3)
1 000	40 ml pure hydrocarbon	40
10	0·4 ml pure hydrocarbon	40
1	10 ml of 1 000 ppm standard mixture	10
0·1	1 ml of 1 000 ppm standard mixture	10

8.1.5 Preparation of C_5 and higher standards requires the transfer of liquids which should be calculated to their volumes as vapour.

$$V_g = \frac{(V_1 \times D \times G \times T \times P) \times 10^3}{(M \times 273 \times 100)}$$

where

V_g = volume in μl of vapour at T and P,
V_1 = volume in μl of liquid at T and P,
D = specific gravity of liquid in g/ml at T,
M = molecular mass in g/mol,
G = gas constant, 22·4 litres/mol at STP,
T = temperature, in K, and
P = absolute pressure in kPa.

8.2 Calibration of Chromatograph

8.2.1 Set instrument parameters at the values listed in **5.6**.

8.2.2 Flush approximately 20 ml of the standard gas through the 1-ml stainless steel sampling loop.

8.2.3 Inject 1 ml of standard gas into the gas chromatograph.

8.2.4 Record the response of the hydrogen flame ionization detector on the strip chart recorder as the hydrocarbon is eluted from the column.

8.2.5 Record the retention time for each hydrocarbon.

8.2.6 Peak height is used for quantitating the C_1 to C_3 hydrocarbons.

8.2.7 Peak area is used for quantitating the C_4 and C_5 hydrocarbons. Peak area is defined as the product of the peak height multiplied by the peak width measured at one half the peak height, as shown in Fig. 4.

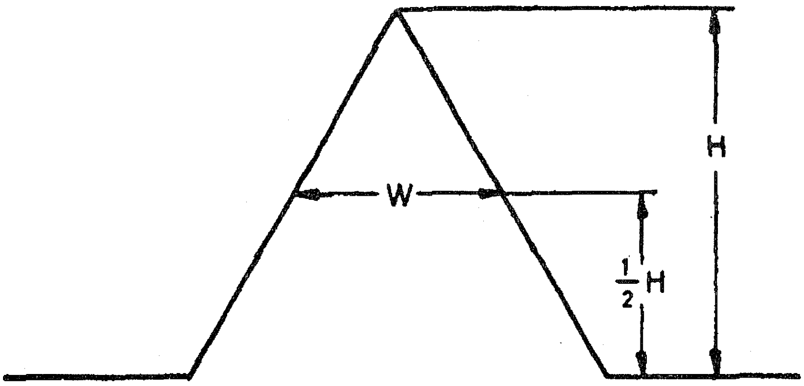


Fig. 4 Measurements for Peak Area

H = peak height,
W = peak width at $\frac{1}{2}$ peak height, and
$H \times W$ = peak area.

Recorder chart speed should be such that peak areas are measureable with a precision of 10 percent. Mechanical or electronic integrators may also be used for peak area measurements.

8.2.8 Calculate a response factor for each hydrocarbon at 10 ppm, 1·0 ppm, and 0·1 ppm. The response factor equals the concentration of the standard in ppm divided by the product of electrometer attenuation and detector response. The detector response for C_1 to C_3 hydrocarbons is peak height measured in millimetres. Peak height is the most convenient measure for quantitation of gases with short retention times when the peaks are narrow and high. The detector response for C_4 and C_5 hydrocarbons is peak area measured in square millimetres. Peak area should be used when peaks are broad and unsymmetrical.

$$f = C/AZ$$

f = response factor, ppm/mm or ppm/mm^2;
C = concentration, ppm;
A = detector response, mm peak height or mm^2 peak area; and
Z = electrometer attenuation.

8.2.9 The response of the hydrogen flame ionization detector is linear from 0·01 to 10 ppm for C_1 to C_5 paraffinic and olefinic atmospheric hydrocarbons.

Note — An error in calculation of peak height or peak area, or in the calculation of instrument response factor may lead to wrong results.

9. PROCEDURE

9.1 The analysis of an air sample without prior concentration involves the procedure given in **9.1.1** to **9.1.7**.

9.1.1 Turn on the recorder.

9.1.2 Set the electrometer attenuation at 10 ×.

9.1.3 Connect the sample bag to the inlet of the sampling valve on the gas chromatograph and flush 20 ml of sample through the 1-ml stainless steel sampling loop.

9.1.4 Inject the sample.

9.1.5 After the elution of methane in approximately 30 s, reset the attenuation to 1 ×. The sample should be eluted from the column in approximately 16 min.

9.1.6 Measure the peak height for C_1 to C_3 hydrocarbons and peak area for C_4 and C_5 hydrocarbons according to the method outlined in **8.2.6** and **8.2.7**.

9.1.7 Calculate the concentration of each hydrocarbon present in the sample according to **10**.

9.2 The analysis of the C_4 and C_5 hydrocarbons to a lower limit of measurement of 0·1 ppb after concentration of the sample involves the procedure given in **9.2.1** to **9.2.7**.

9.2.1 Immerse the freeze-trap 5 cm into liquid oxygen for 10 min with the toggle valves closed and the injection valve bypassing the trap.

9.2.2 Flush a 100-ml gastight syringe three times with the air sample from the aluminized plastics bag and inject 100 ml into the freeze trap at approximately 25 ml/min.

9.2.3 Close the toggle valves on the trap, attach the trap to the injection valve, and vaporize the sample in a water bath at room temperature.

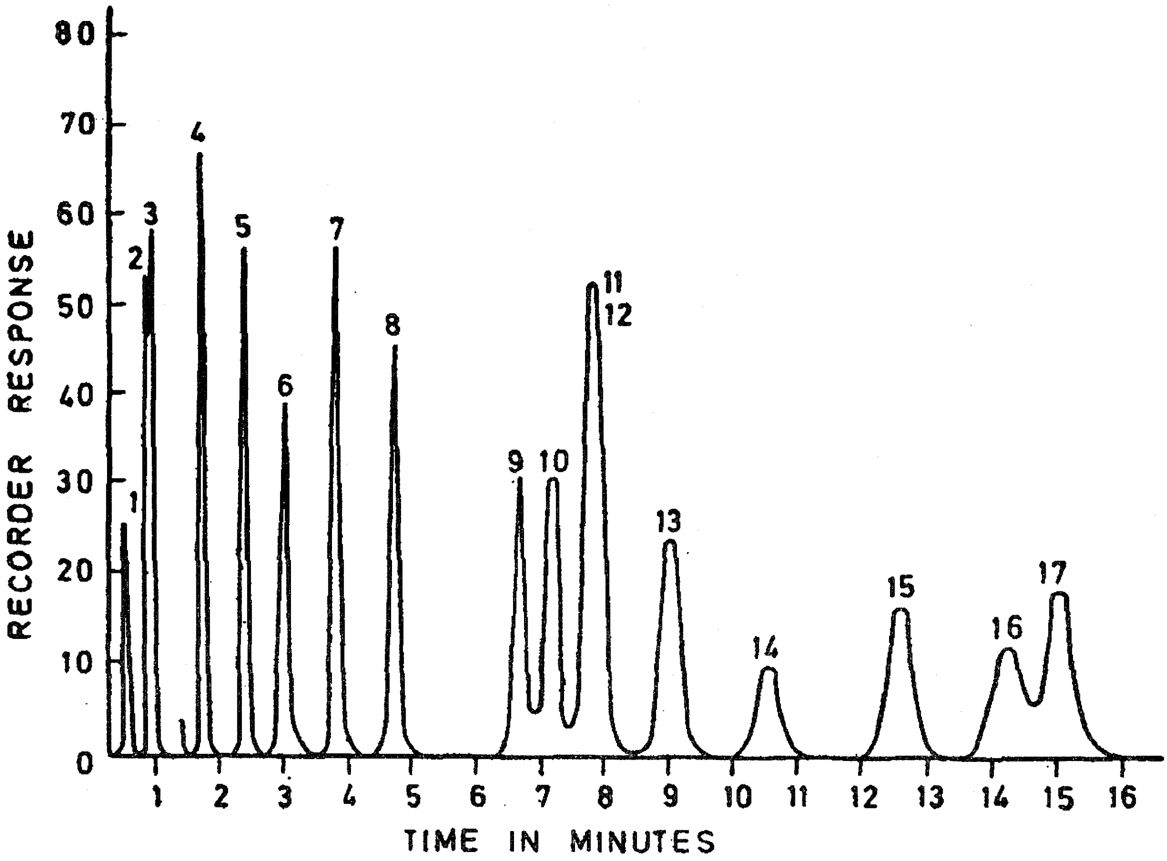
9.2.4 Turn on the strip chart recorder, set the electrometer attenuation at 1 and the output sensitivity at 10 ×.

9.2.5 After a 1-min warm-up of the trap, open in rapid succession the injection valve on the gas chromatograph and the toggle valves on the freeze trap attached to the injector valve.

9.2.6 Measure and identify peaks as stated in **9.1.6** and calculate the concentration of each hydrocarbon as given in **10**.

9.2.7 Unsaturated C_5 hydrocarbons and hydrocarbons of molecular mass higher than C_5 may be eluted after more than 16 min. If these are to be identified and quantitated, calibration for these compounds shall be performed as given in **8** for calibration of the C_1 to C_5 hydrocarbons. If the higher molecular mass hydrocarbons are to be measured routinely, a method producing shorter retention times for them than this low-temperature column may be desirable.

9.3 Figure 5 is a sample chromatogram showing the relative position of the C_1 to C_5 hydrocarbon peaks.



1. Methane
2. Ethane
3. Ethylene
4. Propane
5. Propene
6. Acetylene
7. *Iso*butane
8. *n*-Butane
9. 1-Butene
10. 2-Methylpropene
11. 2, 2-Dimethylpropane
12. *Trans*-2-Butene
13. *Cis*-2-Butene
14. *Iso*pentane
15. 1, 3-Butadiene
16. *n*-Pentane
17. 3-Methyl-1-Butene

FIG. 5 SAMPLE CHROMATOGRAM SHOWING RELATIVE POSITION OF HYDROCARBON ELUTION PEAKS

10. CALCULATION

10.1 The concentration of each of the hydrocarbons in the chromatogram is determined from the response factor for the hydrocarbon, f, the peak response P and the electrometer attenuation, A:

$$\text{Hydrocarbon, ppm} = f \times P \times A$$

where

f = calibration response factor determined in accordance with **8.2.8** for each hydrocarbon in units of ppm/mm of peak height or ppm/mm^2 of peak area;

P = peak height in mm for C_1 to C_3 hydrocarbons or the peak area in mm^2 for C_4 and C_5 hydrocarbons; and

A = electrometer attenuation setting which is generally 10× for methane and 1× for other hydrocarbons.

11. PRECISION

11.1 Retention time and peak response of replicate analyses of an air sample by this method should not deviate by more than 10 percent relative standard deviation.

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